AFOSR-TR-95

0553

# REPORT DOCUMENTATION PAGE



1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
Aug. 16, 1995

3. REPORT TYPE AND DATES COVERED
Final 9/01/94 - 4/01/95

4. TITLE AND SUBTITLE
Sublimation Growth of AlN Single Crystal on AlN coated SiC Substrate

5. FUNDING NUMBERS
~~F49620-94-C-0076~~
63570E
B553/00

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8. PERFORMING ORGANIZATION REPORT NUMBER

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Air Force Office of Scientific Research -NE
110 Duncan Avenue Suite B115
Bolling AFB, DC 20332-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
F49620-94-C-0076



11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Distribution authorized to U. S. Government only

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The technical work of this program resulted in the conclusion that SiC reacted strongly with AlN at high temperature and single crystal AlN was not obtained using AlN coated SiC as seed material. Nevertheless, the undertaking of this program has led to the design and construction of a growth system especially growth crucible and its thermal shielding which eliminates system contamination and corrosion. By using this growth system controllable AlN sublimation-condensation experiments were carried out. Polycrystal AlN discs 20 mm in diameter and up to 4mm thick and single crystal grains and needles 1~3 mm in size were obtained



14. SUBJECT TERMS
Sublimation Growth, Crucible, Shielding, Polycrystal, Single Crystal, AlN, SiC Substrate

15. NUMBER OF PAGES
29

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT
Unclassified

18. SECURITY CLASSIFICATION OF THIS PAGE

19. SECURITY CLASSIFICATION OF ABSTRACT

20. LIMITATION OF ABSTRACT

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)

# F49620-94-C-0076

# Sublimination Growth of AlN Single Crystal on AlN Coated SiC Substrate

## PHASE I

## FINAL REPORT

## August 16, 1995

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## I. EXCUTIVE SUMMARY

This report summarized the progress made on the AFOSR supported program of sublimation growth of AlN single crystal on AlN coated SiC substrate (program number F49620-94-C-0076). The technical works of this program resulted in the conclusion that SiC reacted strongly with AlN coating and source under high temperature sublimation growth condition and single crystal AlN was not obtained using AlN coated SiC as seed. Nevertheless, the undertaking of this program has led to the design and construction of a growth system in which controllable AlN sublimation-condensation process can be studied. Polycrystal AlN disc 20 mm in diameter and 4mm thick were obtained using this growth system.

## II. PROGRAM TASK REVIEW

In this AFOSR supported phase I program our major emphasis was on the use of AlN coated SiC as the seed material to grow bulk AlN single crystals employing sublimation technique. The core tasks embodying this program are the following:

1. Design and build the high temperature furnace for the bulk crystal growth. This includes the design of the reactor, crucible and the heat shield for the crucible. Several alternatives are to be examined.

2. Select suitable source materials, establish the growth parameters needed for the sublimation of the source material. These include the furnace pressure, temperature of the source material and the temperature gradient between the source and the seed.

3. Develop AlN coated SiC seed crystals for the bulk crystal growth. The development includes determining the stability of the thin (1~5 μm) single crystal AlN layer over the SiC substrate. Sublimation procedures are to be developed to avoid dissociation of the seed crystals.

4. Establish sublimation parameters for bulk AlN single crystal growth on AlN coated SiC substrate.

## III. SUMMARY OF TECHNICAL ACCOMPLISHMENTS

During the course of this program we were able to accomplish most of the objectives. The following is a brief description of key technical accomplishments in this program.

We built a AlN sublimation growth system with 20 KW RF output power capable of heating up the growth chamber over 2000°C temperature. We experimented with and found suitable materials and configurations for growth crucible and its thermal shielding which eliminates system contamination and corrosion. This innovative design, for the fist time, allowed us to carry out controlled AlN sublimation-condensation growth without severe system contamination and corrosion. We established sublimation parameters for bulk polycrystal AlN growth. Indeed, we obtained polycrystal AlN discs ~20 mm in diameter and 4 mm thick for one hour growth. We also obtained AlN single crystal grains and needles 1~3 mm in size.

However, due to the strong reaction between SiC and AlN under AlN sublimation conditions, bulk single crystal AlN was not obtained on AlN coated SiC substrate. Free standing (without seed) growth resulted in single crystal needles that may be used as intermediate seeding. Our experiment has also shed some light on finding alternative seeding material for future studies.

## 4. TECHNICAL DETAILS

### 4.1 A brief introduction to sublimation growth

The principle of AlN sublimation growth is based on the chemical reaction shown below which is basically reversible depending on both temperature and pressure of the system:

$$2AlN\ (solid) \Longleftrightarrow 2Al\ (vapor) + N_2\ (gas)$$

That is, at high temperature and low pressure AlN evaporates or dissociates into Al vapor and $N_2$ gas. When the two vapor phases reach their equilibrium partial pressures, the reaction reaches equilibrium state and is reversible if temperature and pressure change towards opposite direction in the system. Inside a system containing Al, $N_2$, and AlN, if one region is hotter than the other, AlN in this region will continue to evaporate and vapor products will move to the colder region and condense there, usually on the two ends of

the chamber which are the coldest spots. From the phase diagram of Al-N, see figure 1, the sublimation temperature of AlN is about 2500°C, which means the vapor pressure of the reaction reaches one atmosphere pressure at this temperature. At 2000°C, the vapor pressure of the reaction is about 100 torr at which AlN sublimation growth may have a proper rate. Therefore, we need a growth system that can reach over 2000°C temperature with controllable ambient pressure.

**4.2 Design of AlN sublimation growth system**

According to the analysis in 4.1 we designed and built a growth chamber with following characteristics:

a. Quartz chamber with 93 mm in diameter and 300 mm high;
b. Water cooling jacket with 1.25 l/min flow rate;
c. Two gas inlets with mass flow controllers;
d. Vacuum bump with pressure controller;
e. Two quartz windows for temperature measuring;
f. A rotating pedestal
g. A movable rf work coil.

Detailed design is shown in figure 2.

**4.3 Design and test growth crucible and its thermal shielding**

The design of the crucible-heating assembly for this high temperature growth has proved non trivial as it should meet the following requirements:

i. Good coupling with rf power and generating enough heat to reach and maintain temperatures over 2000°C. Effective shielding is also necessary.

ii. Crucible, shielding and AlN powder should not react to each other, or diffuse into each other otherwise system contamination and corrosion will result. This also leads to temperature instability.

In fact, only tungsten in the periodic table marginally meets above conditions. But we can not make a efficient shielding using tungsten since the RF coupling to the shields is not desirable. This is the biggest obstacle in AlN sublimation technique. We have tried many combinations to solve this problem. Table 1 and 2 show the materials and sizes of the crucibles and shielding materials used in our experiments for finding suitable combinations of crucible and shielding. Following is a summary of our experiment sequence.

a. Graphite crucible and graphite felt as used in SiC sublimation growth. Figure 3 illustrates the basic setting of SiC sublimation system. The advantage of this combination is very good in coupling with rf power and maintaining high temperature as used in SiC sublimation growth (~2500°C). The problem is that Al vapor reacts with carbon strongly and form $Al_4C_3$ or AlCN as soon as AlN evaporates. The resulting substances wet and cover the AlN source and the desired sublimation growth of AlN is impossible.

b. Graphite crucible, graphite felt and sintered AlN cavity. This combination has the same problem as in a. AlN was contaminated by carbon. But inside AlN cavity, the AlN sublimation-condensation condition was achieved and small rod or platelet of AlN single crystal was formed.

c. Small CVD tungsten crucible set and graphite felt. The crucibles with pointed tip which designed for free seeding growth. Because of the small size of the crucible, the coupling with the rf power was poor. The temperature was far below required growth condition. .

d. Tungsten foil sleeve and graphite felt. Tungsten foil sleeve with suitable size can couple with rf power reasonably and reaches desired high temperature. The small thermal mass of the thin foil does however cause temperature non-uniformity. In addition the thin tungsten foil degraded quickly in presence of Al vapor and carbon specie from graphite felt. But sufficient sublimation-condensation processing was observed. Needles and grains of AlN crystal grew on the colder part of tungsten sleeve. The problem of this combination is still carbon contamination from graphite felt.

e. Tungsten foil sleeve and alumina felt. In nitrogen and at high temperature (about 1600°C) conditions, alumina felt became hardened and darkened, the shielding efficiency degraded. Therefore, it is not a suitable shielding for AlN sublimation growth.

f. Tungsten foil sleeve and zirconia felt. In nitrogen and at high temperature (about 1400°C) conditions, zirconia was reduced by nitrogen and a brown smoke emitted from the felt. We also used porous zirconia cylinder as shielding. Same phenomenon occurred. Therefore, it can not be used as shielding in AlN sublimation growth.

g. Tungsten foil sleeve and alumina tube. Alumina tube was not efficient to shield heat from dispersion, therefore, the growth temperature can not reach required condition.

h. Tungsten crucible and tungsten foil disc and gold inside coated chamber as reflecting shielding. The advantage of this shielding is that there is no contamination, but it is not efficient enough to allow us to reach the growth temperature.

i. Tungsten foil sleeve, BN liner and graphite felt. BN liner was attacked and destroyed by carbon at high temperature.

j. Tungsten foil sleeve and AlN powder held by a quartz beaker. The detail configuration is shown in figure 4. The top temperature of the tungsten foil cap was 1800 to 2000°C. After one hour growth, the AlN power outside the tungsten foil sleeve sintered into an egg-shaped crust embracing the sleeve. Inside this crust, the dimension of the hollow is greater than that extended by the tungsten foil sleeve. The source material (AlN sintered chops) loaded inside the tungsten sleeve was transferred to the top-inside of the crust forming an olive-shaped polycrystal AlN as illstrated in figure 5. This result indicated that AlN sublimation and condensation process had occurred and progressed for a period of time. The conditions experienced inside the crust were close to what required for AlN sublimation growth.

k. Tungsten foil sleeve, tungsten disc and AlN sintered crucible, AlN powder, quartz beaker, the detailed configuration of the system was shown in figure 6. The growth conditions were 600 torr nitrogen pressure, 1850°C, 2000°C for top and bottom temperatures, respectively. The growth time was one hour. A polycrystal AlN disc ~20 mm in diameter and 2 to 4 mm thick was grown on a tungsten disc as shown in figure 7.

l. With a 3 mm thick CVD tungsten crucible, two thick tungsten discs and a machined AlN sintered crucible, AlN powder, a quartz beaker, the modified design was similar to that shown in the figure 6. Unfortunately the high frequency rf generator was not coupled with this CVD tungsten crucible as well as it did with tungsten foil crucible. The temperature only reached to 1600°C at maxima power output of 20 KW rf generator..

In summary, we have built and tested many combinations of crucible and shielding sets. The best of all was summarized in k and the corresponding configuration was shown in figure 6. The results of above tests also indicate that by using this growth system and testing parameters controllable AlN sublimation-condensation process can be achieved.

**4.4 Temperature distribution of the growth system.**

A cylindrical tungsten foil crucible with small thermal mass can be heated up above 2000°C by means of rf heating. However, the temperature inside crucible was not uniform, especially at the beginning of experiments. Our many experimental efforts were focused on obtaining repeatable temperature profile inside crucible. One way to change temperature distribution is to move the work coil up or down relative to the tungsten crucible. By setting crucible height properly in the chamber and adjusting work coil height during the growth process, the temperature readings from top window and bottom window can be adjusted at will. But please note that these two readings only show two coldest spots of the crucible and the temperature profile inside crucible is basically unknown. We defined

the top reading as the growth temperature and the bottom one as the source material temperature. The movable work coil configuration and an example of temperature reading were shown in figure 8. After many experiments we found that the better temperature profile is a positive temperature gradient from bottom part to top part of the crucible (i.e. $T_{bottom} > T_{top}$ ). The top opening of the crucible should be sealed by AlN powder completely during growth. In this way the temperature profile inside crucible is relatively uniform. The temperature gradient mainly depends on the relative height of the work coil to the tungsten crucible.

**4.5 Establish AlN sublimation growth parameters**

For systematically studying the behavior of AlN sublimation and condensation process we have made following set of experiments in the crucible-shielding configuration similar to that shown in figure 6. Note that inside crucible, crystal growth occurs both on tungsten cap and top part of the source material. We defined them as growth on tungsten cap and free standing growth respectively. We set the growth conditions as follows, growth (top)temperature: from 1600°C to 2000°C; Temperature gradient: 100~200°C; Nitrogen pressure: 76, 300, and 600 torr respectively. For many reasons, it is difficult to control the growth temperature precisely. We have to use actual readings for particular experiments. The results were presented in Table 3 and 4. Following is a summary:

a. Sublimation rate of AlN depends both on temperature of source material and ambient nitrogen pressure, the higher the temperature and the lower the pressure, the faster the AlN sublimation rate. Feasible sublimation starts from 1600°C. Above 2100°C AlN sublimes very fast under 76 torr nitrogen pressure.

b. AlN growth rate also depends both on temperature and nitrogen pressure, but the relationship is more complicated, because low nitrogen pressure enhances both the sublimation of AlN which increases growth rate and the escape of sublimed vapors from the crucible which decreases growth rate. Temperature gradient plays an important role on the growth rate. Large temperature gradient results in more free standing growth. Negative temperature gradient results in no growth but source material sintering. Our experiments also indicated that stable growth behavior of AlN may need higher than atmosphere pressure of nitrogen.

c. AlN crystal morphology is clearly related to growth temperature. When growth temperatures were at and below 1700°C, white felt like AlN crystal grew and loosely attached onto tungsten foil cap. This white deposit was actually fine fibers of AlN, see figure 9. When growth temperatures were between 1800 and 1900°C, brown colored

polycrystal AlN disc grew on tungsten cap. The grain size depends on growth temperature, the higher the temperature, the larger the grain size, ranging from 0.1 mm to 1 mm. Meanwhile, the crystal color became darker as temperature increases. Short needles or rods were found as free standing growth crystal at these growth temperatures, see figure 10(a) and (b). When growth temperatures reached to and above 2000°C, single crystal grains grew on the wall of tungsten foil crucible with the size of 1~3 mm in diameter. The individual grains grew separately and the facets of the crystal were well developed. Figure 11 show the crystal grains taken off from the crucible. No free standing growth found under this condition. In general, the morphology of the grown crystal did not change abruptly with temperature.

d. Suitable sublimation growth conditions. As mentioned before, low growth temperature results in fiber or needle growth. This implies that a higher growth (seed) temperature, i. e. above 2000°C should be used and the suitable source material temperature should be correspondingly higher, i. e. 2100 to 2200°C. From our results, the sublimation rate at this temperature would be too high at less than one atmosphere pressure. Therefore, the suitable pressure conditions should be in the range of 1 to 100 atm.

**4.6 AlN sublimation growth on AlN coated SiC substrate**

Three types of SiC substrates have been used in this experiment those are Lely crystal, Achson crystal and CREE crystal. An AlN coating about 1 μm thick was deposited on SiC substrates in a low pressure MOCVD reactor using TMA (trimethylaluminum) and ammonia as Al and N sources respectively. Resulting AlN films were examined by HREED and shown single crystal structure. The growth configuration was shown in figure 12. SiC substrate supported by a ring of sintered AlN and placed just beneath tungsten cap. AlN particles loaded as source material. The results were presented in Table 5. Following is a brief description.

a. When growth temperatures were at or below 1600°C and 600 torr nitrogen pressure, no growth of AlN found on SiC substrate but the surface was etched slightly, see figure 13. Note that the SiC substrate was AlN coated before sublimation growth.

b. When growth temperature was at 1700°C and 300 torr nitrogen pressure, for 0.5 hour growth, white AlN fine fibers about 1 mm thick was loosely deposited on SiC growth surface. While the back surface was etched.

c. When growth temperature was at 1800°C and 600 torr nitrogen pressure, for one hour growth, about 0.1 mm thick polycrystal AlN was grown on the substrate. The grain size was about 0.05 mm. On the edges of the substrate no growth but substrate surface was etched. These were shown in figure 14(a) and (b).

d. When growth temperature was kept at 1800°C but nitrogen pressure was lowered to 76 torr, little AlN growth found on growth surface of SiC substrate, instead, the surface was etched, hexagonal terraces was formed on the substrate surface. The back surface of the substrate was also etched and Si droplet with different sizes left on it. When increasing nitrogen pressure to 600 torr the silicon drops disappeared gradually. Figure 15(a) and (b) show the morphology of both sides of the SiC substrate after growth at 1800°C and 76 torr.

e. When growth temperature raised to 1900°C, and at 150 torr nitrogen pressure, after one hour growth, no growth found on SiC substrate. Instead, the thickness of SiC substrate reduced to a quarter of the original one and some black deposits found on tungsten cap behind SiC substrate. A micrograph of substrate surface was shown in figure 16.

In summary, polycrystal AlN can be deposited on SiC substrate at 1800°C, but at higher growth temperature, Al vapor reacts with SiC strongly and no deposits formed on SiC surface, instead, SiC etching rate increases rapidly.

**4.7 Conclusion**

we have established reasonable sublimation growth conditions for AlN bulk growth. Small single crystals of AlN in needle or grain shape with the size of 1~3 mm have been obtained. Polycrystal AlN discs of 20 mm in diameter and 4 mm in thickness have been grown on polycrystal tungsten disc repeatably. SiC substrate coated with AlN thin film is not a good candidate as AlN single crystal seed due to strong reaction between AlN and SiC at high temperature.

Table 1 A list of crucibles used in this program

1. Graphite crucible, high density 0.5"OD x 0.375" ID x2" H
2. Graphite crucible, high density 1"OD x 0.6" ID x2" H
3. Graphite crucible, high density 1.25"OD x 1" ID x2.5" H
4. Graphite crucible, high density 2"OD x 1" ID x2" H
5. Machined tungsten crucible 1"OD x 0.5" ID x3" H
6. Tungsten foil crucible 1 mil thick, 0.5~2" OD x2~6"H
7. Tungsten foil crucible 2 mil thick, 1~2" OD x2~4"H
8. Tungsten foil crucible 5 mil thick, 1.25~2" OD x1.5~2.5"H
9. Tungsten foil crucible 10 mil thick, 2" OD x2.5"H
10. Tungsten disc 5 mil thick, 1.4" OD
11. Tungsten disc 30 mil thick, 1.4" OD
12. CVD tungsten crucible 1.4"OD x 1.25" ID x2.5" H
13. CVD tungsten crucible set including
    2 conical crucibles 0.25"OD x 0.18" ID x0.6" H
    up sleeve crucible 0.4"OD x 0.25" ID x1.25" H
    bottom sleeve crucible 0.5"OD x 0.4" ID x1.4" H

Table 2 A list of shielding materials used in this program

1. Graphite felt, high purity, 0.125" thick
2. Alumina felt, no bonding organic, 0.125" thick
3. Zirconia felt, high purity, o.125" thick
4. Alumina sleeve, high purity, 2"OD x 1.75"ID
5. Zirconia cylinder, high purity, 2"OD x1"ID
6. Tungsten foil reflecting shielding 1 mil thick
7. Gold coated quartz chamber
8. AlN fine powder
9. Sintered AlN crucible 2"OD x 1.45"ID x 4" H
10. BN crucible
11. BN fine powder

Table 3 A list of parameters used in AlN sublimation growth

| | | |
|---|---|---|
| I<br>Tg= 1600-1700 C<br>Pres. =600 torr | IV<br>Tg= 1700-1850 C<br>Pres. = 600 torr | VII<br>Tg= 1850-2000 C<br>Pres. = 600 torr |
| II<br>Tg= 1600-1700 C<br>Pres. = 300 torr | V<br>Tg= 1700-1850 C<br>Pres. = 300 torr | VIII<br>Tg= 1850-2000 C<br>Pres. = 300 torr |
| III<br>Tg= 1600-1700 C<br>Pres. = 76 torr | VI<br>Tg= 1700-1850 C<br>Pres. = 76 torr | IX<br>Tg= 1850-2000 C<br>Pres. = 76 torr |

Note: Tg: growth temperature, temperature gradient 100~200 C, Pres. nitrogen pressure.

Table 4 A summary of AlN sublimation growth results

| No | AlN Source | Growth on cap | Free standing growth | Reference |
|---|---|---|---|---|
| I | Not sintered together, stick on crucible | white powders, <0.2 mm thick | Fine fiber, d~0.005 mm | Run# 53, 54, 55, 130, 133 |
| II | slight sintered at two end | White powder ~0.5 mm thick | More fine fibers; d~ 0.005 mm | Run # 126, 127 |
| III | Slight sintered together | White powder 1-3 mm thick | Thin hex needle d~ 0.02 mm | Run# 141, 142, 143 |
| IV | Sintered together, gap <1 mm | Brown deposit, 0.2-2 mm thick | Needles, DxL~0.2 x 4 mm | Run# 31, 50, 87 |
| V | Sintered hard gap ~1 mm | Dark brown deposit 1-3 mm thick | Short needles, DxL~0.5x3 mm | Run# 125,132 |
| VI | Sintered hard, gap >5 mm | Dark brown deposit 1-3 mm thick | A few grains, d< 0.5 mm | Run# 85, 87 |
| VII | Half AlN gone | grains on crucible d~1-3 mm | Form d~15 mm polycrystal olive | Run# 18, 26 |
| VIII | Half AlN gone | Dark deposit ~3 mm thick | No free standing growth | Run# 129 |
| IX | Half AlN gone | Dark deposit ~3 mm thick, ~5 mm | No growth, outside crucible ~5 mm AlN sintered hard, brown | Run# 144 |

Table 5 A summary of AlN sublimation growth on AlN coated SiC substrates

| Run number | Growth condition | SiC substrate | Growth | Comment |
|---|---|---|---|---|
| 1.2 | Tg=1700 C, 600 torr, 30 min. | etched at edges | poly AlN ~0.07 mm | in graphite crucible |
| 4.2 | Tg=1650 C, 600 torr, 20 min. | growth surface etched | no growth | AlN film coated on SiC |
| 29.4 | Tg=1700 C, 600 torr, 50 min | thinned, a big Sidrop at back | Poly AlN grains, D~0.2 mm | temp. measured on AlN cap |
| 36.4 | Tg=1400 C, 600 torr, 30 min | white deposit on back | Poly AlN grains, D~0.2 mm | temp. measured on AlN cap |
| 48.4 | Tg=1900 C, 600 torr, 60 min. | disapeared | ~0.5 mm deposit on W cap | some AlN source blacked |
| 49.4 | Tg=1600 C, 600 torr, 30 min | etched back face | many fiber growth | |
| 68.4 | Tg=1220 C, 600 torr, 30 min. | both sides etched | some poly AlN | temp. measured on AlN cap |
| 89.4 | Tg=1280 C, 200 torr, 50 min | back surface etched | white deposit | temp. measured on AlN cap |
| 93.4 | Tg=1330 C, 150 torr, 60 min. | back etched with Si drop | etched w/o Si drop | temp. measured on AlN cap |
| 95.4 | Tg=1622 C, 150 torr, 70 min. | etched to 1/4 thick | no growth | |
| 125.4 | Tg=1700 C, 300 torr, 30 min. | back side etched | white fiber ~1 mm | AlN film coated |
| 132.4 | Tg=1840 C, 300 torr, 30 min. | back side etched | white deposit ~ 1mm | AlN film coated |
| 140.4 | Tg=1600 C, 76 torr, 30 min. | back side slightly etched | thin white deposit, edges etched | AlN film coated |

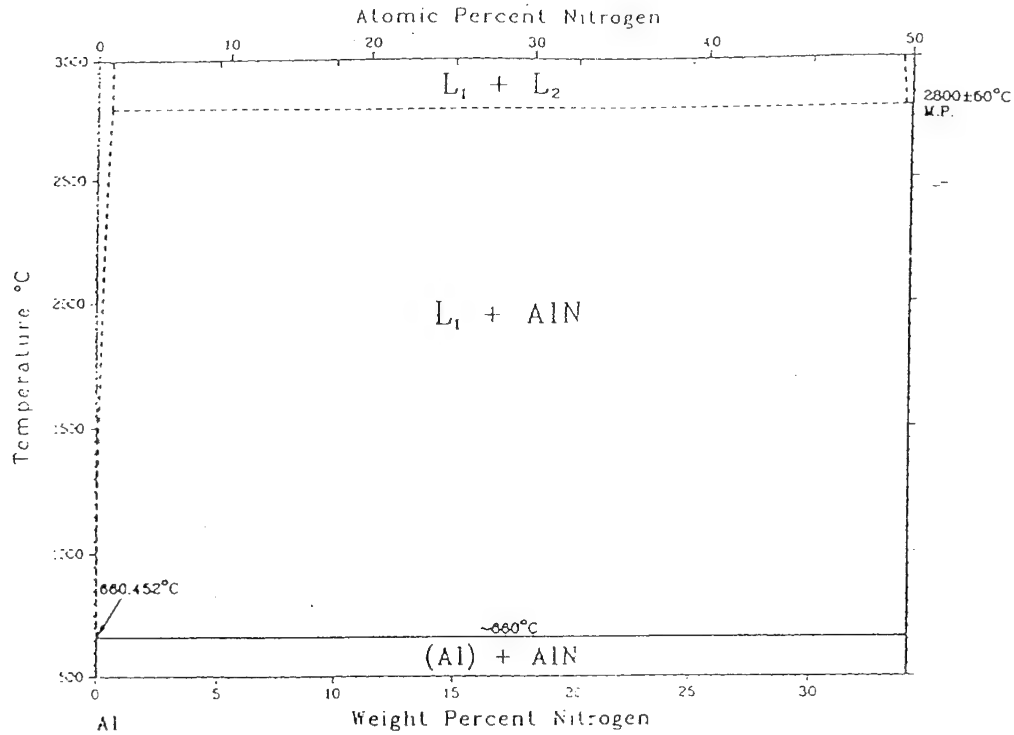



Figure 1(a) Phase diagram of Al-N system (from "Binary Alloy Phase Diagrams" Vol 1, Editor in Chief T. B. Massalski)

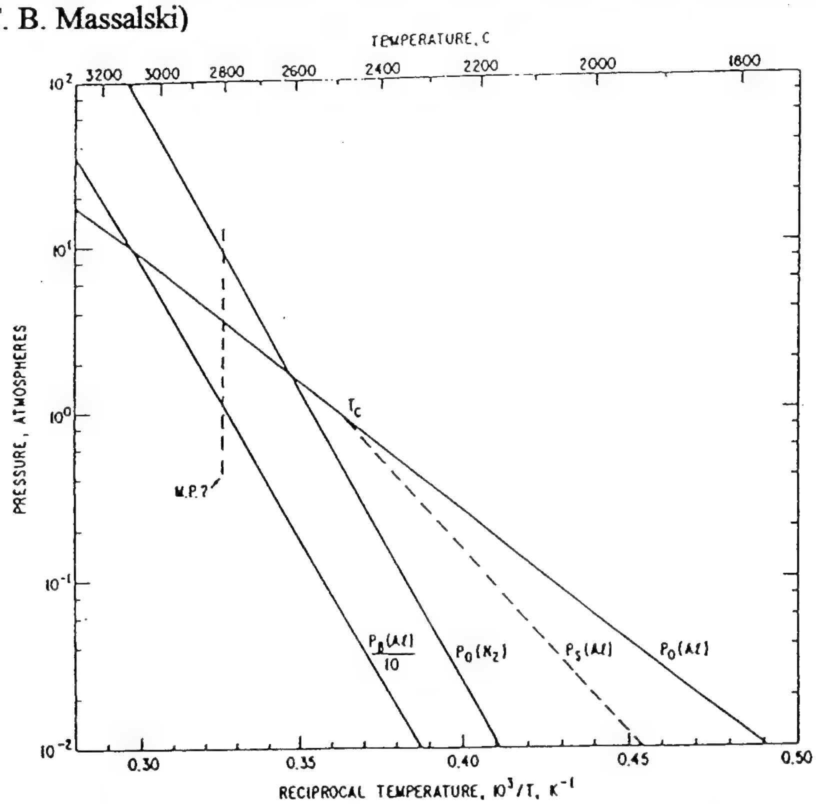



Figure 1(b) Temperature-pressure (T-P) plot of Al-N system (from G. A. Slack and T. F. McNelly "Growth of high Purity AlN Crystals", J. Crystal Growth, **34**. (1976) 263-279

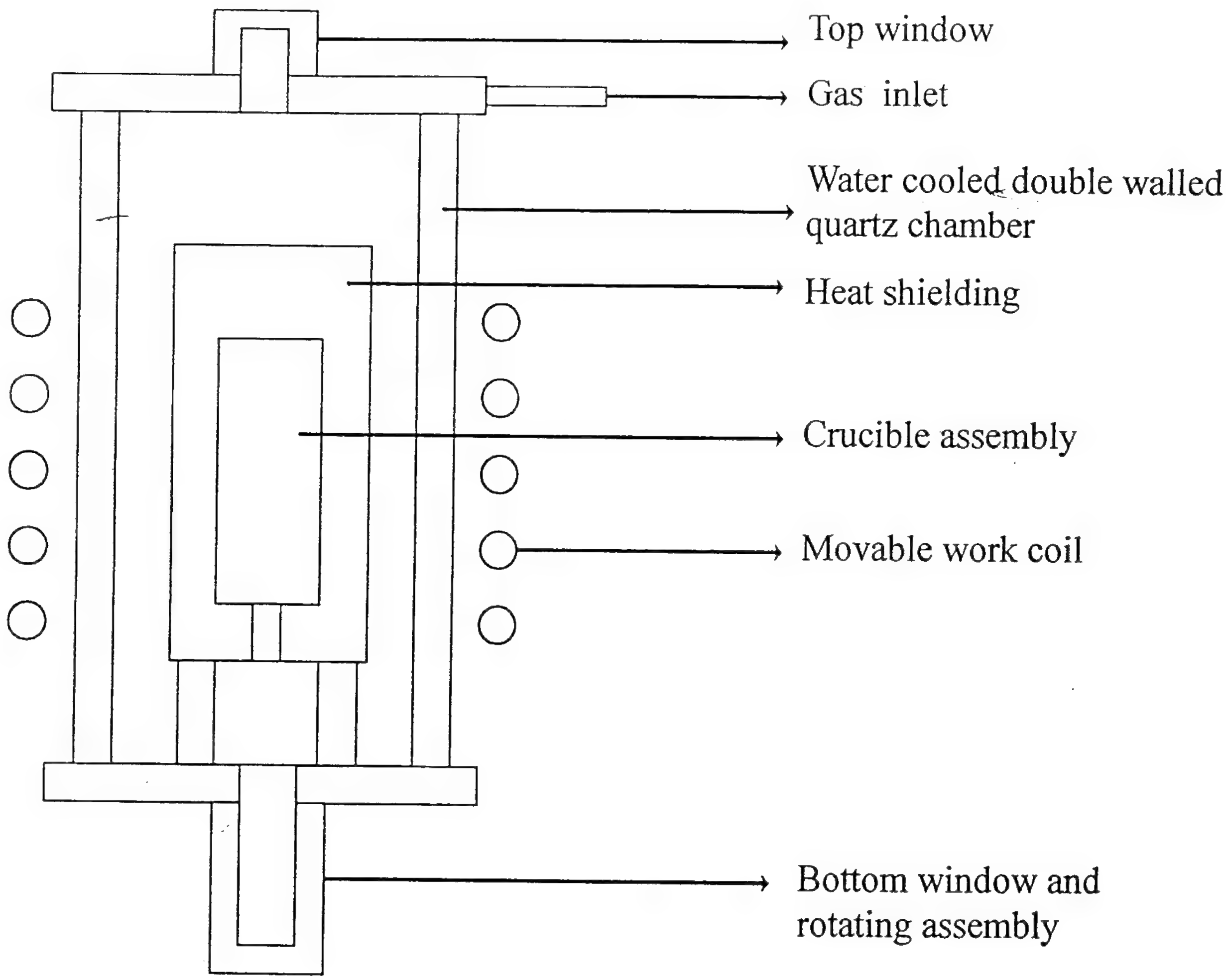



Figure 2 Schematic of AlN sublimation growth system

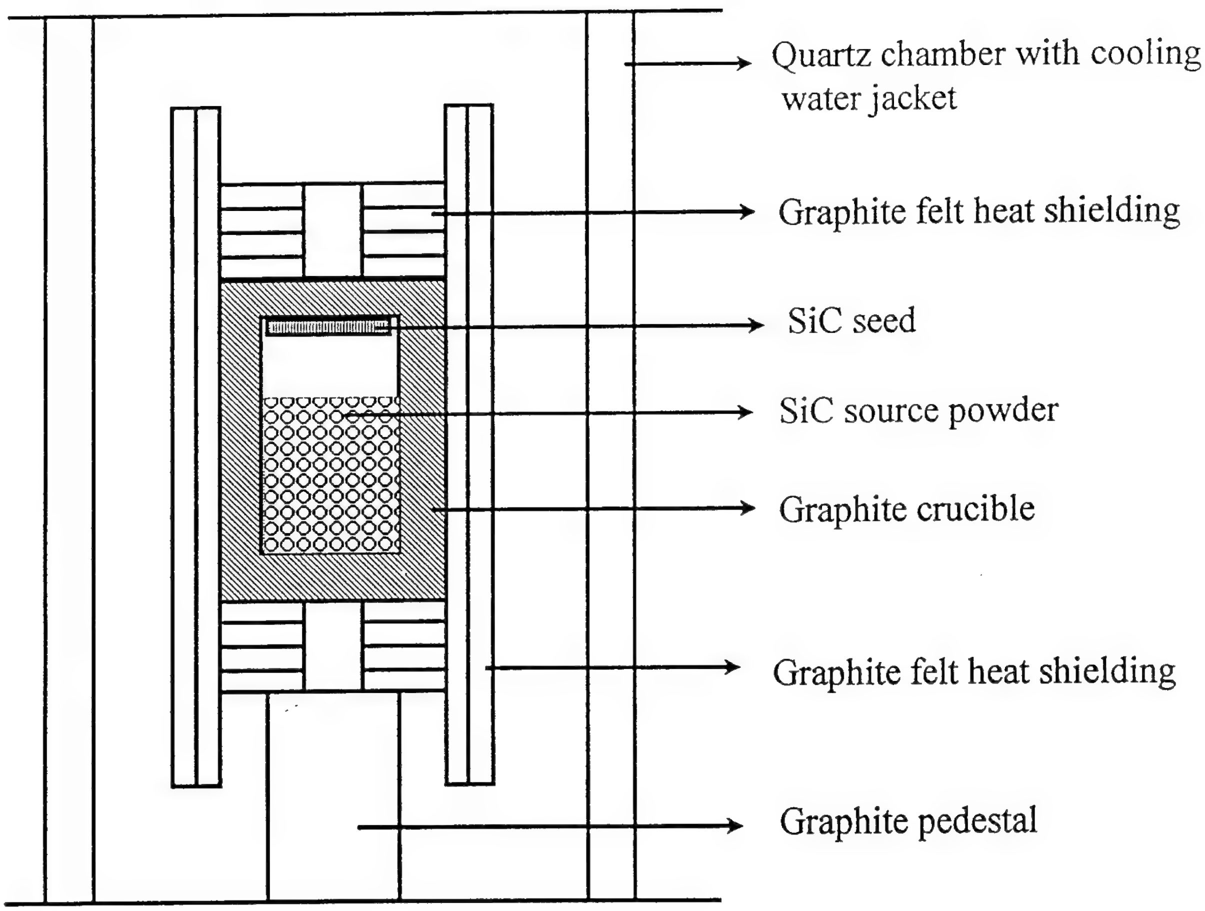



Figure 3 Schematic of SiC sublimation growth system

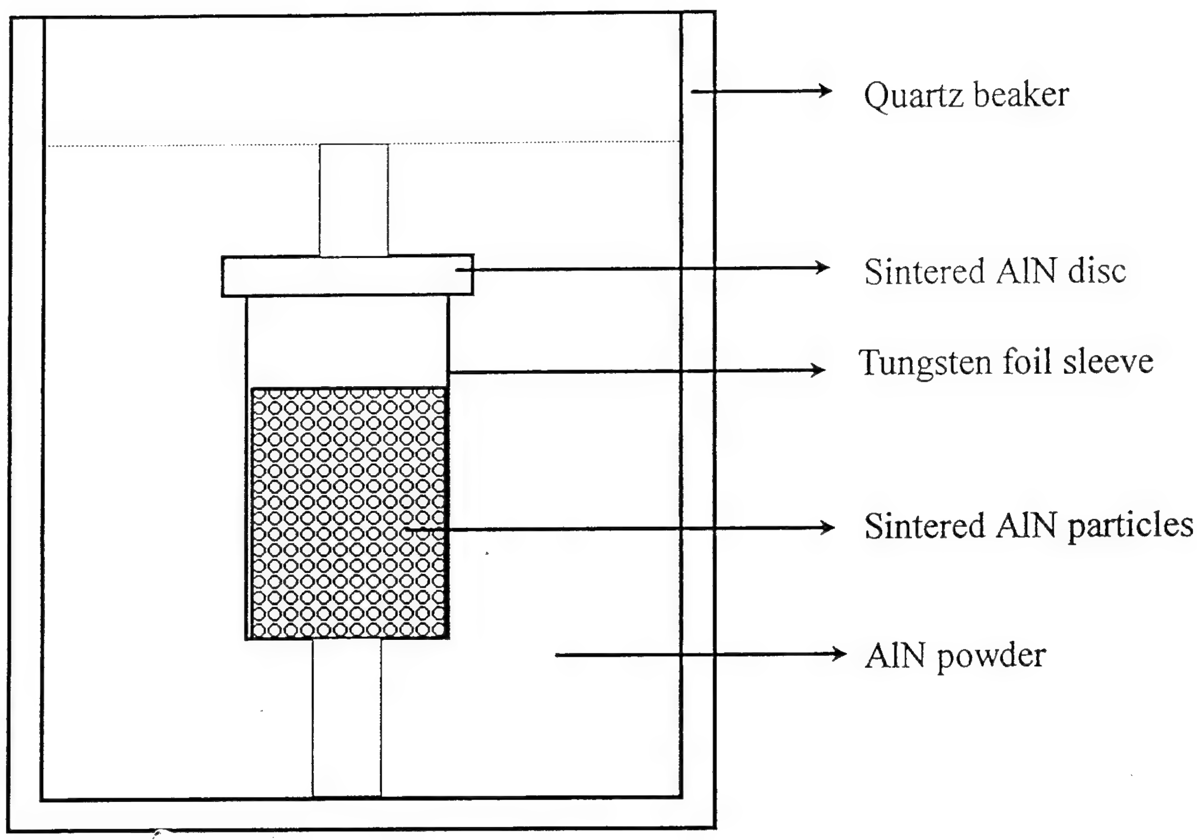
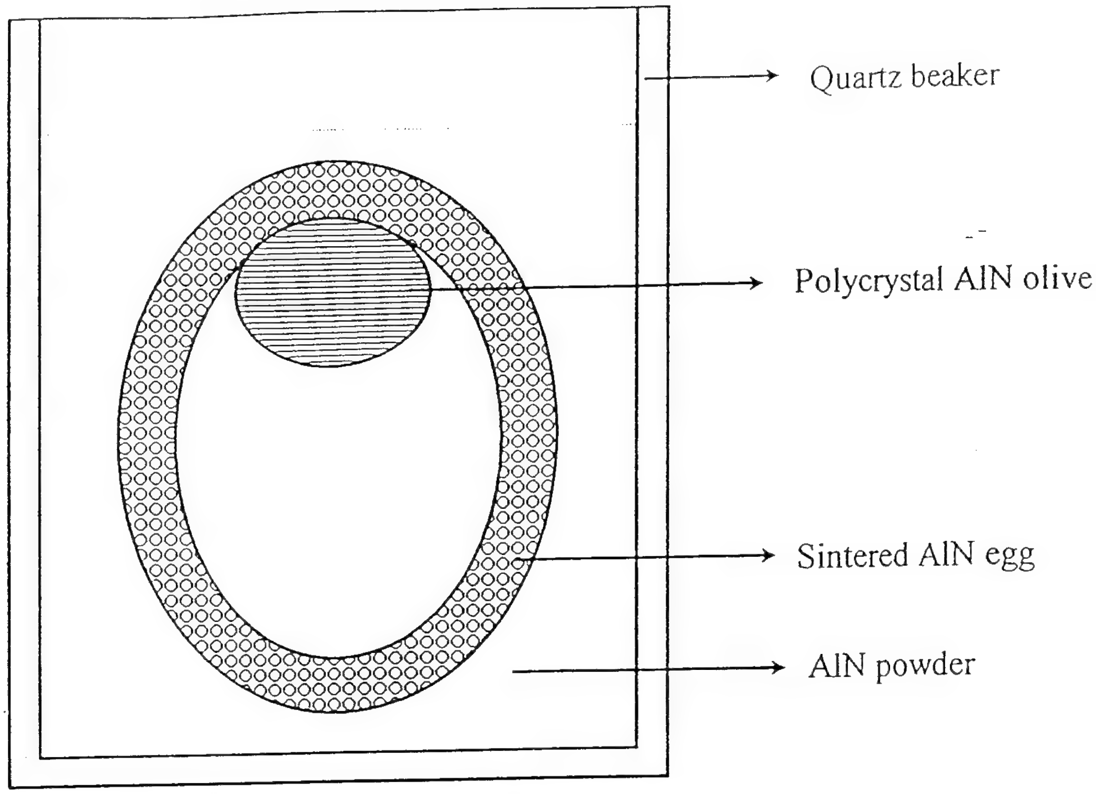



Figure 4 Configuration of a crucible-shielding set using tungsten foil sleeve, AlN powder, and a quartz beaker.



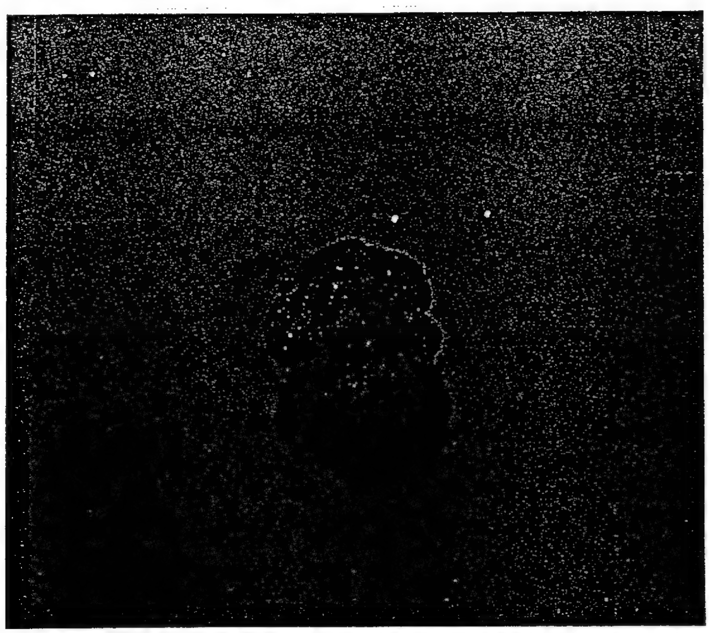



Figure 5(a) A schematic of an olive shaped polycrystal AlN formed in a sintered AlN crust. 5(b)A photograph of olive shaped polycrystal AlN (x1.2).

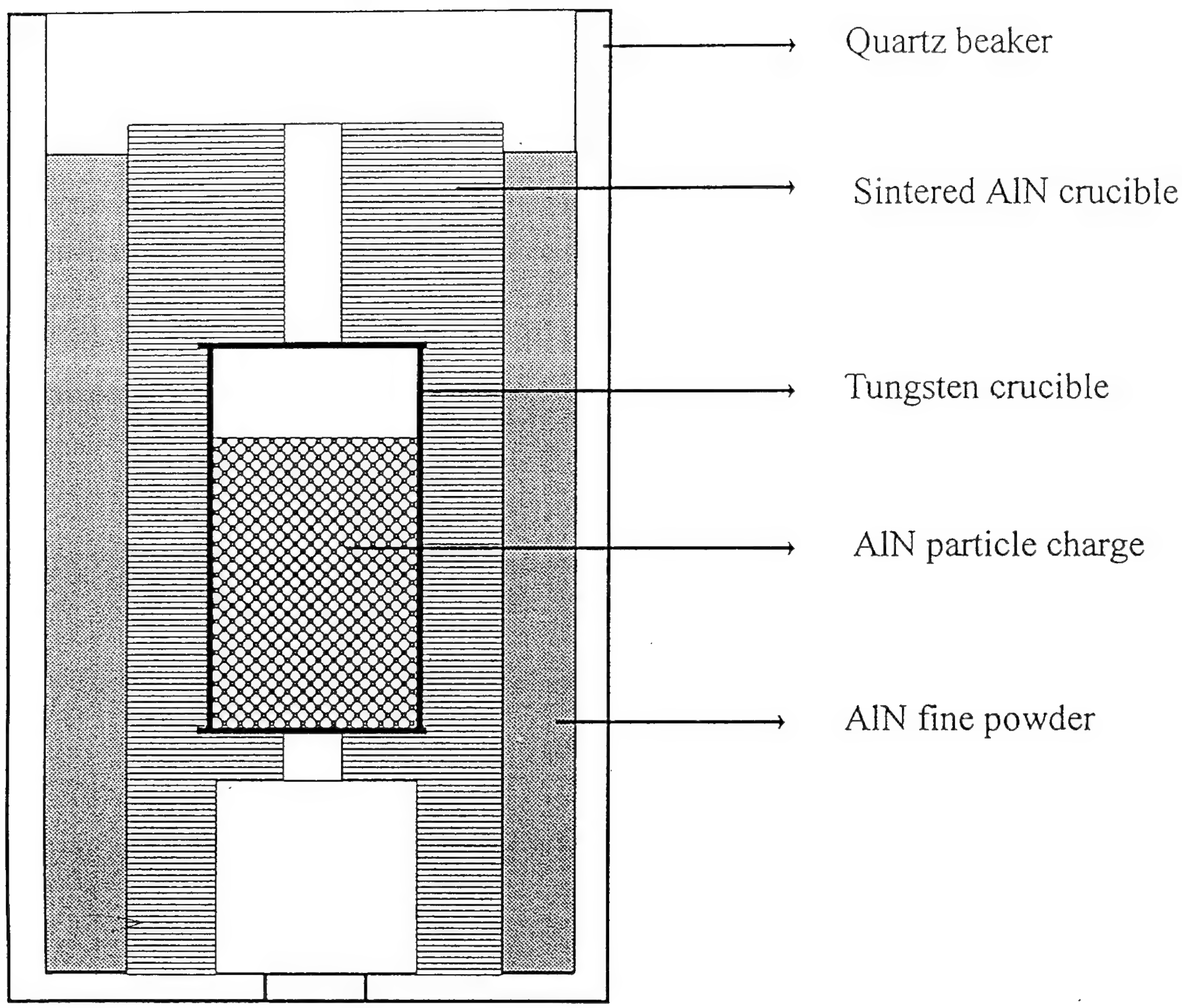



Figure 6 Final configuration of crucible-shielding set using tungsten foil sleeve as heating crucible, tungsten discs as crucible caps, sintered AlN crucible as outer crucible, AlN fine powder as insulating material, and a quartz beaker as structural support.

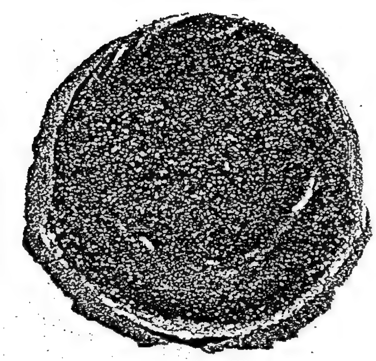
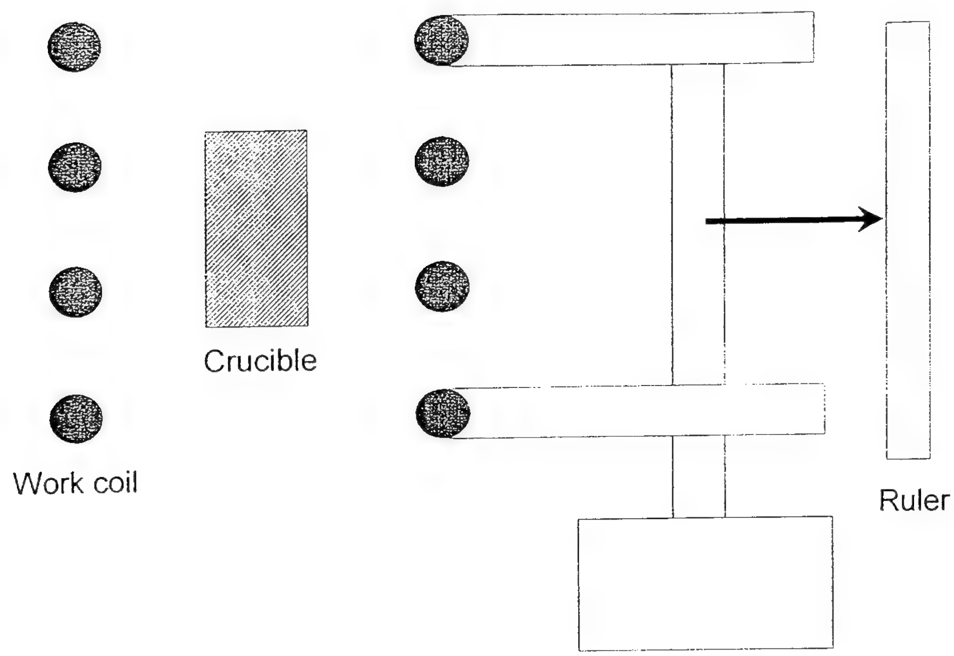


Figure 7 A photograph of a polycrystal AlN disc grown on a tungsten cap (x1.5).




| Top temperature C | Bottom temperature C | Work coil height mm |
|---|---|---|
| 1550 | 1700 | 0 |
| 1650 | 1650 | 12 |
| 1700 | 1500 | 25 |

Figure 8 An illustration of movable work coil in the growth system and a table of temperature reading vs work coil height.

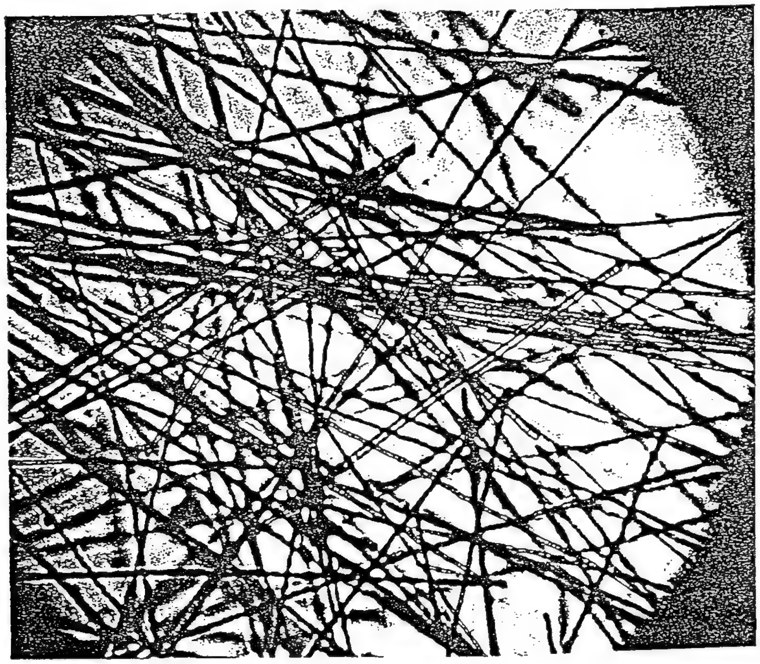
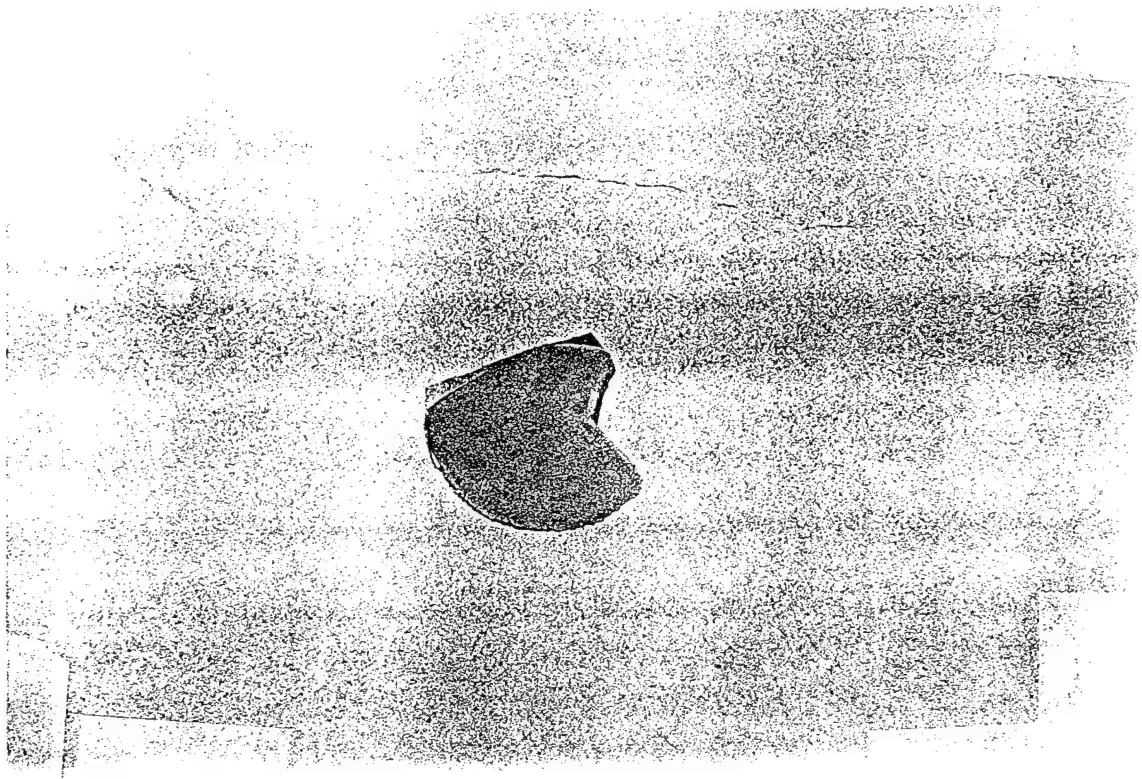


Figure 9 A photograph of fine AlN fibers grew on the top tungsten cap at 1700$^{o}$C for one hour (x200).



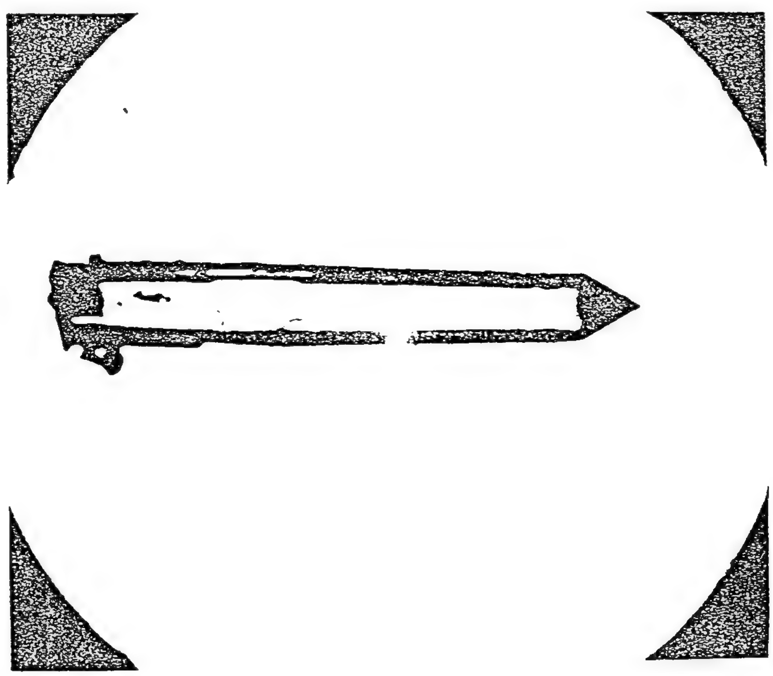


Figure 10(a) Photographs of a polycrystal AlN disc grown on a tungsten cap(x1.5) and 10(b) a short needle found on top of AlN source at 1850$^{o}$C(x100)

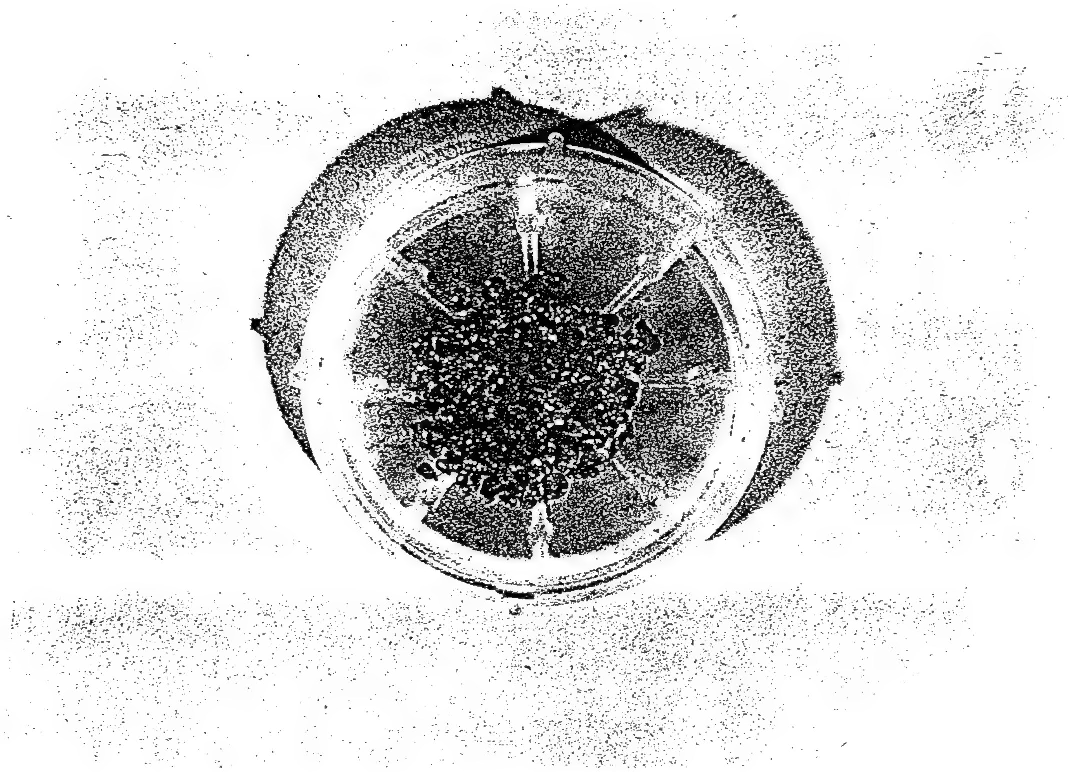


Figure 11 A photograph of single crystal AlN grains and needles taken off from tungsten foil crucible wall after growth process above 2000$^{o}$C(x1.5).

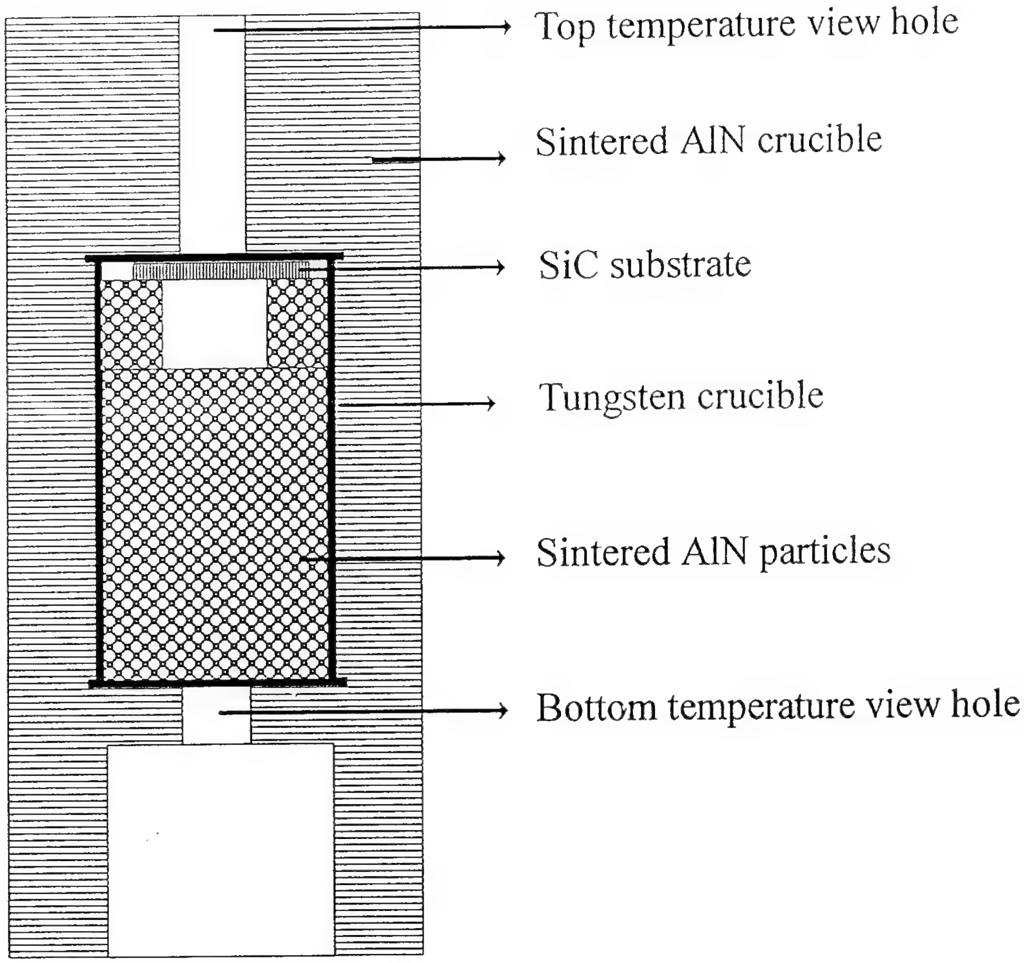



Figure 12 The configuration of AlN sublimation growth using AlN coated SiC as seed material.

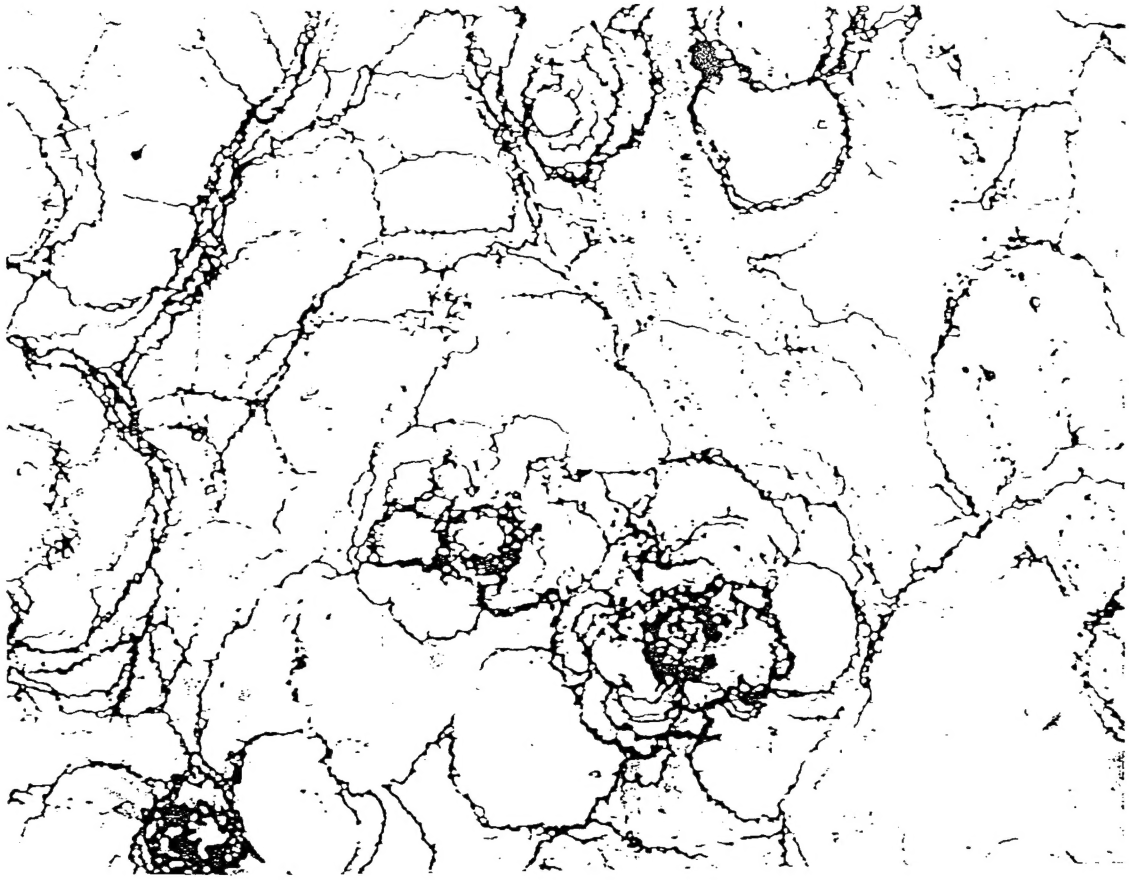


Figure 13 A photograph of AlN coated SiC substrate surface after AlN sublimation growth at 1600°C for 1 hour. The coating surface was slightly etched (x400).

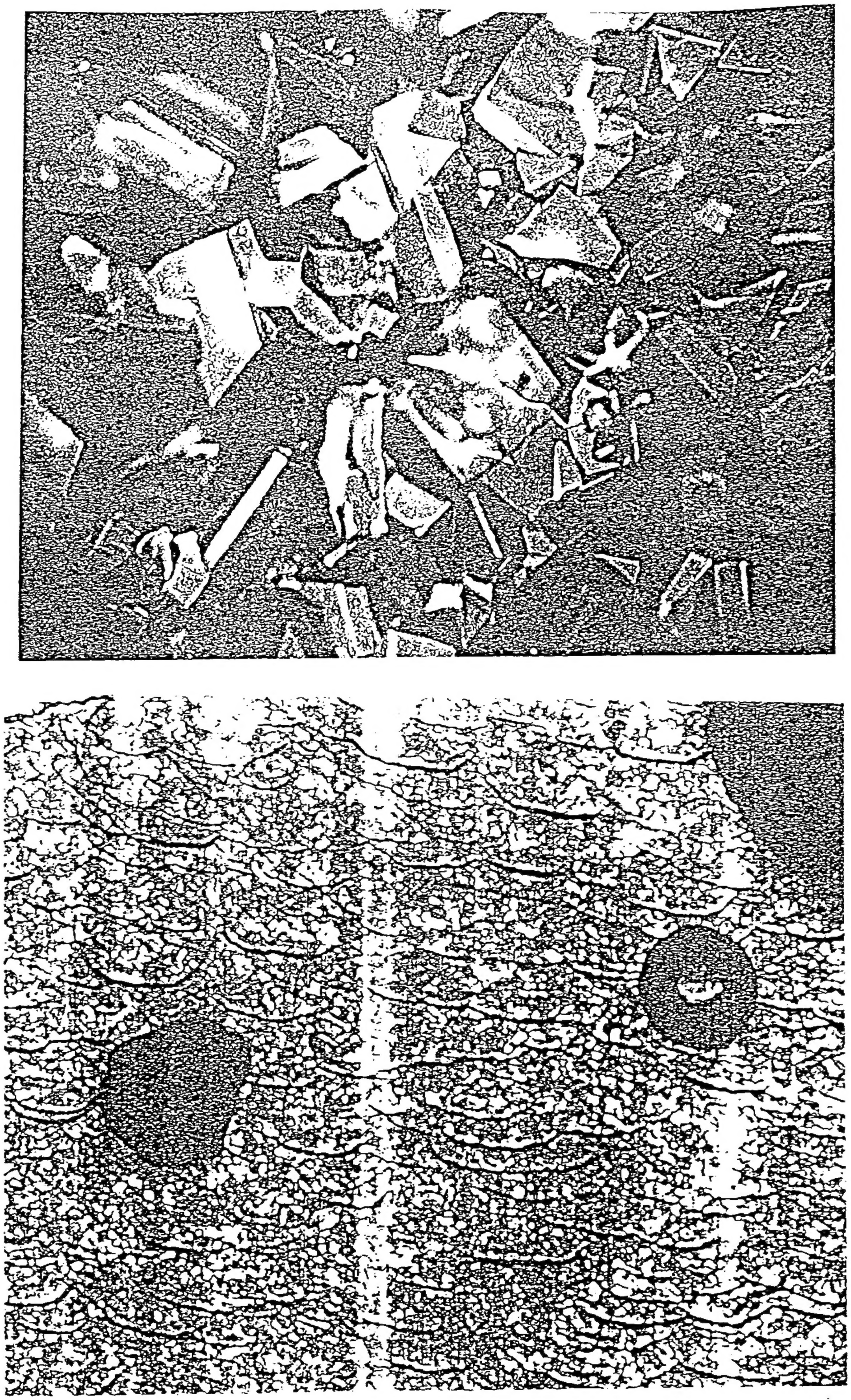


Figure 14 Photographs of polycrystal AlN layer grown on the central part of AlN coated SiC substrate (a), no growth but etched on the periphery of the substrate (b). both x400

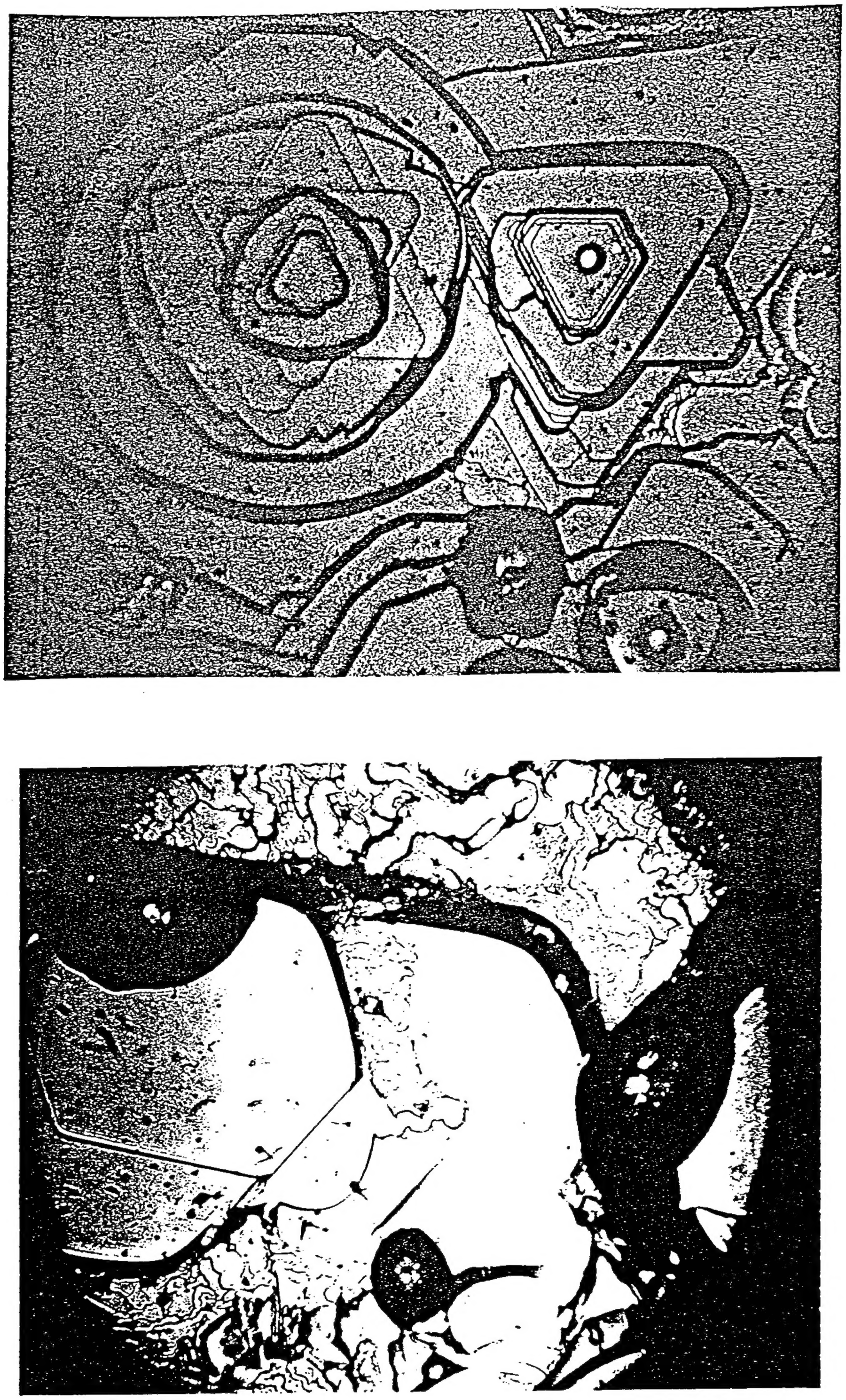


Figure15 Photographs of SiC growth surface (a), no growth found but hexagonal etching terraces. The back surface of the substrate was also etched and Si droplet left on it (b) after one hour growth at 1800$^{o}$C and 76 torr nitrigen preasure(both x200).

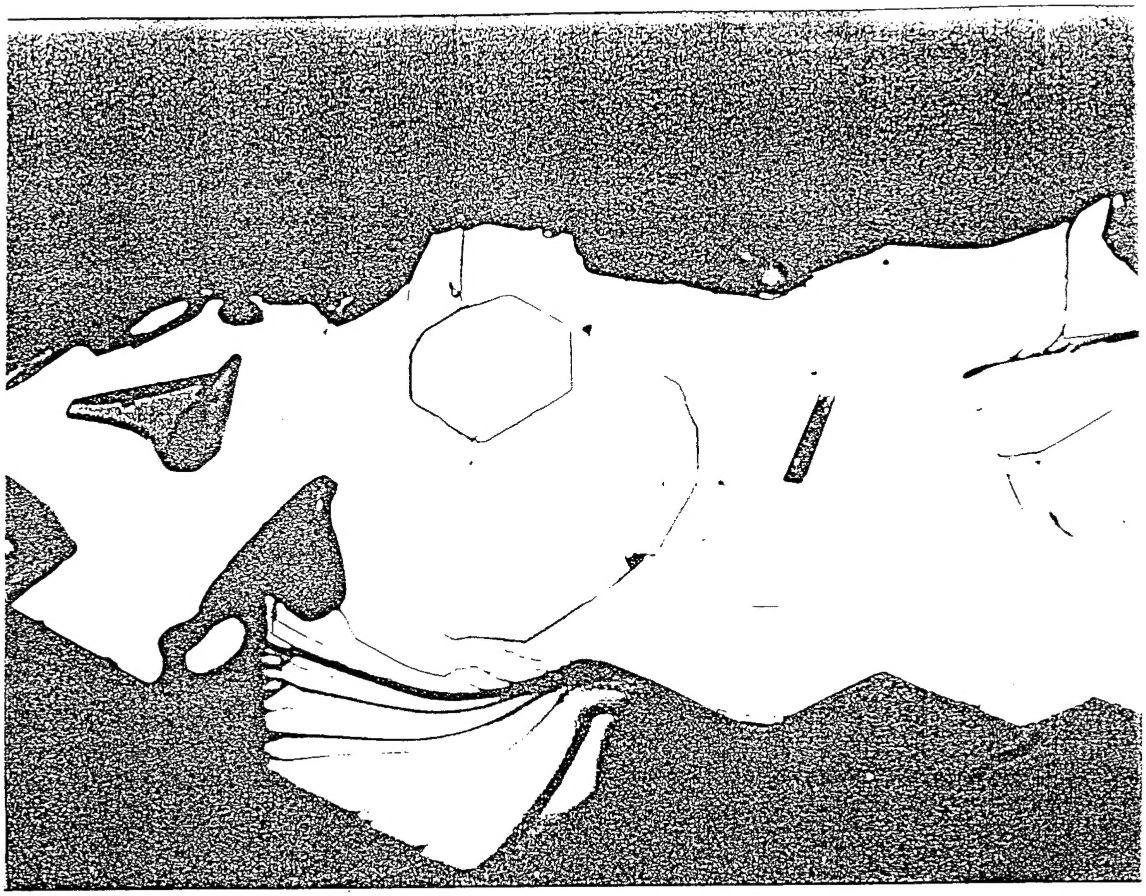


Figure 16 A photograph of residual SiC substrate about a quarter of the original thickness left on tungsten cap after one hour growth at 1900°C and 600 torr nitrigen pressure (x375).